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8. AUTHOR(s)

Amiya K. Mukherjee, Giora Gurewitz and
Mike Meier.

9. PERFORMING ORGANIZATION NAME AND ADDRESS

Division of Material Science
Department of Mechanical Engineering
University of California, Davis, CA 9561610. PROGRAM ELEMENT, PROJECT, TASK
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The mechanical and microstructural aspect of superplastic deformation in Ti-6Al-4V alloy was investigated with special emphasis given to cavitation phenomenon. The contribution of grain growth and grain refinement in affecting the flow stress at a given temperature and strain rate was noted. Cavities nucleate and grow mostly at temperature below 850°C. Above 900°C cavity nucleation was almost nonexistent due to higher volume fraction of the soft deformable phase in the microstructure.

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**FUNDAMENTAL INVESTIGATIONS ON FAILURE
DURING OF SUPERPLASTIC FORMING PROCESSES**

**Annual Technical Report Coving the Period
Between 2/1/82 and 1/31/83 ..**

Submitted by

Professor Amiya K. Mukherjee, Giora Guerwitz and Michael Meier

Division of Materials Science and Engineering

Department of Mechanical Engineering

University of California

Davis, California 95616

to

Dr. Alan Rosenstein

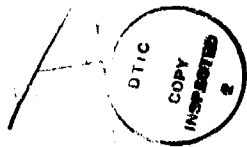
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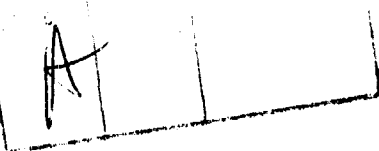
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I INTRODUCTION

Coupled with diffusion bonding, the superplastic forming operation has allowed airframe manufacturing engineering to a more cost effective design with significant savings in materials cost, labor intensive machining cost, and the possibility of obtaining large geometrically complicated shapes in one step. The remarkable surface finish, isotropic mechanical properties and saving of energy during forming operation are added advantages to the superplastic forming process.

Although most attention has been devoted to the flow behavior of superplastic materials, another important behavior has received very little attention. It is the area of fracture in superplastic materials. It was thought for many years that internal cavities do not form during superplastic deformation. However, it is now known that cavitation may be an important process even in materials exhibiting large superplastic ductility.

The interest in the problem of fracture in superplastic materials arises for the following reasons:

(a) The need to reduce the extent of internal cavitation in superplastic alloys of commercial significance--alloys based on Al, Cu, Ni, and Fe demonstrate serious cavitation problems. Even the Ti-6Al-4V alloys, that have found extensive application in air frame parts cavitate (1) at lower forming temperatures. The extent of cavitation in this alloy was shown to increase (2) sharply with a small increase in grain size.

(b) Cavitation at high temperature leads to problems of intergranular embrittlement in many high-strength, high temperature alloys when subject

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to creep, low-cycle fatigue, welding and thermal cycling. A detailed study of cavitation in superplasticity may assist in better understanding of one of the most serious current fracture problems.

(c) In some superplastic forming operations, it is not the maximum attainable ductility but the extent of strain dependent cavitation that sets a limit to the operating parameters to successful forming operations.

It is, therefore, important to understand the factors that lead to cavitation during superplastic deformation. A better understanding of these factors will eventually enable us to devise procedures which minimize the occurrence of cavitation. The current proposal outlines a three-year experimental as well as theoretical approach to provide detailed information on the influence of stress, strain, strain rate, temperature and grain size or phase size on the fracture characteristics of two superplastic material systems.

It is anticipated that studies of microstructural changes, mechanical behavior, fracture surfaces and density measurements will eventually shed enough light to enable us to describe the cavity formation and interlinkage behavior in terms of reasonable constitutive expressions for fracture. A successful implementation of such studies should provide the tools to construct three-dimensional fracture mechanisms maps for specific superplastic material systems.

SUMMARY OF PRESENT STATUS UNDERSTANDING ON SUPERPLASTIC CAVITATION AND FAILURE

(a) There has been some progress in our understanding of cavitation and fracture process in superplasticity. However, many basic issues remain

unclarified. During superplastic deformation in tension, some alloys cavitate extensively before fracture, often the cavities starting at fairly early strain. Cavitation occurs during tensile flow but not during purely compressive flow. In general, in many alloy systems, the volume of cavities increased with increasing strain. The dependence on strain rate and temperature is ambiguous and can vary from one alloy system to another. Superplastic cavitation can arise from the localization of flow along the grain and interphase boundaries by the process of grain boundary sliding. Hard particles can act as stress concentrators during the sliding process.

(b) There are substantial and significant differences in the details of cavitation in creep and that in superplasticity. Apart from the very important factor of the smallness of grain size and the much enhanced level of grain boundary sliding, superplastic cavitation is also greatly affected by the strain rate sensitivity of the material. In superplastic alloys, their propensity to retain large tensile elongations has been shown to be related to the delayed coalescence of cavities, rather than to any reduced rate of nucleation or growth. The stability of the deformation of the ligament region between cavities is enhanced by the increased strain rate sensitivity associated with superplastic flow of the bulk material.

(c) The theoretical understanding of cavitation and fracture in superplasticity is on far less firm ground than similar understanding of creep failure. The constitutive expressions for superplastic cavitation is very inadequately developed at present. This problem is compounded by the fact that there is a great scarcity of good experimental data (mechanical, microstructural and physical data e.g., density measurements, etc.) on well characterized materials system that fail by superplastic cavitation.

The growth of a cavity in superplasticity may be controlled by vacancy diffusion or power-law creep. In general, diffusional growth is favored at low total strains and there is a transition to power-law growth at a critical cavity radius. Currently available theoretical estimates show that whereas the trend in transition from diffusion to power law growth with increasing cavity radius is correct, the estimated size of the critical radius at transition is seriously in error.

The final stages of cavitation are closely related to catastrophic neck growth. In exploring the problem of cavity coalescence, the question need be addressed as to whether the rate sensitive properties are also scale sensitive. Presumably, at cavity spacing less than one grain diameter, the properties are indeed sensitive to scale features. More work yet need be done in this aspect in order to relate the cavitation phenomenon ultimately to actual failure stress.

2. PROGRESS DURING CURRENT PROGRAM YEAR

2.1 ABSTRACT

During the past year, we have initiated an experimental research program in order to study the nucleation and growth of cavities in Ti-6Al-4V alloy as a function of temperature, strain-rate and strain. We have used scanning and transmission electron microscopy and quantitative metallography, coupled with differential as well as constant (and continuous) strain rate tests. We have established the shape of true stress and true strain curves at four strain rates for each of three constant temperatures. Clear evidence of cavity nucleation and growth

have been observed. The extent of cavitation increases as temperature decreases and cavitation is very easily discernible at 750°. Above 925°C cavitation is not easily detectable.

During the same period we have reviewed the relevant literature (including our previous work with the alloy) and have extracted the pertinent parameters for the rate controlling mechanism for superplasticity in Ti-6Al-4V alloy. Using these parameters in the elevated temperature constitutive expressions for various creep mechanisms and superplasticity, we have constructed two and three dimensional deformation mechanism maps from available experimental data from literature.

We have also investigated the details of the stress-strain curves during deformation of Ti-6Al-4V in order to understand the role of grain growth leading to strain hardening and grain refinement (primary through dynamic recrystallization) leading to strain softening.

In a separate series of investigations the stress-strain rate characteristics of Ti-6Al-4V at different temperatures are being studied. The activation energy for superplastic deformation and the dependence of this apparent activation energy on microstructural activation are also being investigated.

2.2 Summary of Progress

Mechanical testing

All the experiments were conducted on a MTS servohydraulic machine interfaced with a PDP/11 computer. This enabled us to use the computer both as a controller in order to conduct either constant strain rate tensile tests or differential strain rate tests. The computer also

interfaced with a digital data acquisition system. The latter system allowed us to collect, store, recall and evaluate the mechanical results and to subsequently plot the results in desired coordinates.

We are thankful to AFOSR for approval in earlier years for the partial purchase cost of this system. Without this help we would not have the flexibility in our mechanical testing that we have today.

The Quad Elliptical Radiant Heating Furnace provided a heating rate of 200°C/min. The phaser power controller gave excellent temperature-control capability ($\pm 1^\circ\text{C}$). The tests were conducted in an atmosphere of purified argon gas.

The Ti-6Al-4V alloy was supplied by TIMET corporation. After vacuum casting the ingot was forged in ' β ' phase and subsequently hot rolled at 925°C to give a total reduction of 97 percent. This thermomechanical processing produced a very fine grained equiaxed grain structure.

RESULTS

The mechanical tests were conducted at 750°, 800°, and 850°C. At each temperature, the specimens were tested using four different strain rates: 10^{-4} , 5×10^{-5} , 2×10^{-5} and 1×10^{-5} per second. These combination of test temperature and strain rates assured the fact that the specimen was deforming in the typical region II of superplasticity. At any of these constant strain rate tests, the experiment was stopped at a preprogrammed true strain of 0.2, 0.4, 0.6, 0.8, 1.0 and 1.2 and the specimen was quenched in pre-chilled argon gas and cooled under load. This was done in order to preserve the elevated temperature deformed microstructure for subsequent scanning and transmission microscopy observations.

Figures 1, 2, and 3 show the true stress vs. true strain curves for 750°, 800°, and 850°C respectively. At 750° and 800°C, at the two highest strain rates investigated, the curves show evidence of strain softening. Preliminary TEM studies suggests that this is due to dynamic recrystallization (Fig. 12). The two slowest strain rate curves at 750°, 800°, and 850°C show clear evidence of strain hardening. This we believe is due to the observed grain growth.

The volume fraction of " β " phase (as observed in the quenched and tested specimens at room temperature) is shown in Fig. 3. as a function of strain. It is seen that whereas the volume fraction of β phase is unaffected as a function of strain at 750° and 800°C, it is a sensitive function of strain at 850°C. We also have noted a significant difference in the characteristics of cavity appearance above and below 850°C. We believe that these two observations are linked and we intend to pursue this further. At present it appears that higher is the volume fraction of the soft deformable β phase, lower is the incidence of cavitation.

A typical result of differential temperature tests is given in Fig. 5. In these experiments a single specimen is strained at constant temperature at various strain rates in either an incremental or decremental fashion. From the slope of a plot of $\log(\text{stress})$ vs. $\log(\text{strain rate})$ one evaluates the strain-rate sensitivity parameter " m ". The m -values in ideal superplastic condition is typically around 0.5. Our estimated values of m -parameter from experimental results were $m = 0.53$, 0.54, and 0.66 for 850°, 800°, and 750°C respectively, which is close to what we expect in superplasticity.

Microstructural Observations

The tested specimens were investigated for evidence of cavity growth using both SEM and TEM. Fig. 6 shows the SEM micrograph of a specimen tested at 750°C at 10^{-4} s^{-1} strain rate up to a true-strain $\epsilon = 1.05$. It reveals the presence of a cavity between two (darker) alpha-phase grains. Fig. 6 is transmission photograph showing an elliptical shaped cavity at the junction of three grain boundaries. Fig. 7 shows multiple cavities at the grain boundaries at 800°C for specimens tests to a true strain $\epsilon = 1.3$.

Deformation Mechanism Maps

Stress, strain-rate and temperature are the three most important parameters in the high temperature deformation process, i.e., creep, hot rolling hot-forging, superplastic processing etc. The various elevated temperature micromechanisms for plastic flow are getting fairly well established now. We have already produced two and three dimensional deformation mechanism maps using such principles for the Al-Zn alloy system.

Using the method described in earlier progress report of our AFOSR sponsored work, we have constructed two and three dimensional deformation mechanism maps for Ti-6Al-4V alloy. The stress and grain size dependence of strain rate and the activation energy were gathered from available experimental data in the literature. The pre-exponential constant 'A' was estimated by fitting the stress-strain rate data from current tests. The procedure is described in "Two and Three Dimensional Maps for the High Temperature Creep of Zn-22 percent Al Alloy", A. Arieli and A. K. Mukherjee, Materials Science and Engineering, 47, (1981), 113.

Fig. 9a and 9b shows the stress-strain rate relationship for constant grain size and constant temperature situation respectively. Fig. 10. shows

the two-dimensional deformation mechanism maps for either stress-temperature relationship at different (but constant) grain size or the stress-grain size relationship for different (but constant) temperature. Finally Fig. 11. shows the three-dimensional deformation mechanism map as a function of normalized-stress, grain size and temperature. Here region III is dislocation creep, region II is superplasticity and region I is a distinct but as-yet an unidentified mechanism.

In a new series of investigations with Ti-6Al-4V, we are trying to establish from primary experimental data the following parameters

- (a) the stress dependence of strain rate
- (b) the dependence of strain rate on instantaneous (i.e., strain enhanced) grain growth
- (c) the activation energy of the deformation process

(d) the value of pre-exponential constants in the rate equations for superplasticity and creep. Item (b) is important because grain growth and particularly strain enhanced grain growth is an important aspect of microstructural change in Ti-6Al-4V. It raises the value of flow stress at any given temperature and strain rate. Its effect is more pronounced at low strain rate and higher temperature. By investigating the strain, temperature and strain rate dependence of the grain size, we hope to compensate for the deformation enhanced grain growth in superplasticity. By incorporating the instantaneous grain size, we hope to arrive at more realistic values for the rate parameters for the constitutive equations for superplasticity and creep. This inturn should enable us to produce more dependable 3-dimensional deformation mechanism maps for this alloy.

The following is a short description of the outcome of this preliminary investigation. Fig. 13 shows the effect of strain rate at a constant temperature of 925°C. Fig. 14 shows the effect of temperature at a constant strain rate of $2 \times 10^{-4} \text{ s}^{-1}$. One observes strain softening at higher strain rates or lower temperature. Similarly at lower strain rates or higher temperature one observes strain hardening (due to grain growth). Figs. 15, 16, and 17 depict the log flow stress-log strain rate relationship at the onset of yielding, at 0.2 true strain and at 0.5 true strain respectively. The rest of needed data points are being generated at present, Fig. 18 to Fig. 21 shows the experimentally measured activation energy at the onset of yield, and at 0.2, 0.5, ~0.75 strain respectively. The activation energy decreases with increase in strain (Fig. 22). This is undoubtedly due to microstructural alterations which are being investigated at present.

2.3 Conclusion

a) We have noticed cavitation in superplastic deformation only at the grain boundaries or phase boundaries and at grain boundary triple-points. At present it appears to be always so.

b) It appears from present work that the strain hardening, during superplastic deformation at lower strain rates is due to grain growth and the strain softening at higher strain rate is due to dynamic recrystallization and the consequent refinement of grain size. Transmission electron microscopy supports this conclusion.

c) We recall the fact that the volume fraction of β phase (Fig. 4) increased substantially as a function of strain at 850° (but not at 750° and 800°C) and the fact that above 900°C there was very little noticeable cavitation. It appears that an increased volume fraction of β phase minimizes the probability of cavitation by making the accommodation process due to grain boundary sliding easier.

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5. A. Arieli and A. K. Mukherjee, "High Temperature Creep of Zn-22 percent Al Eutectoid Alloy. Part I - Mechanical Behavior," to be presented at 109 AIME Annual Meeting, Las Vegas, Nevada, February 1980.
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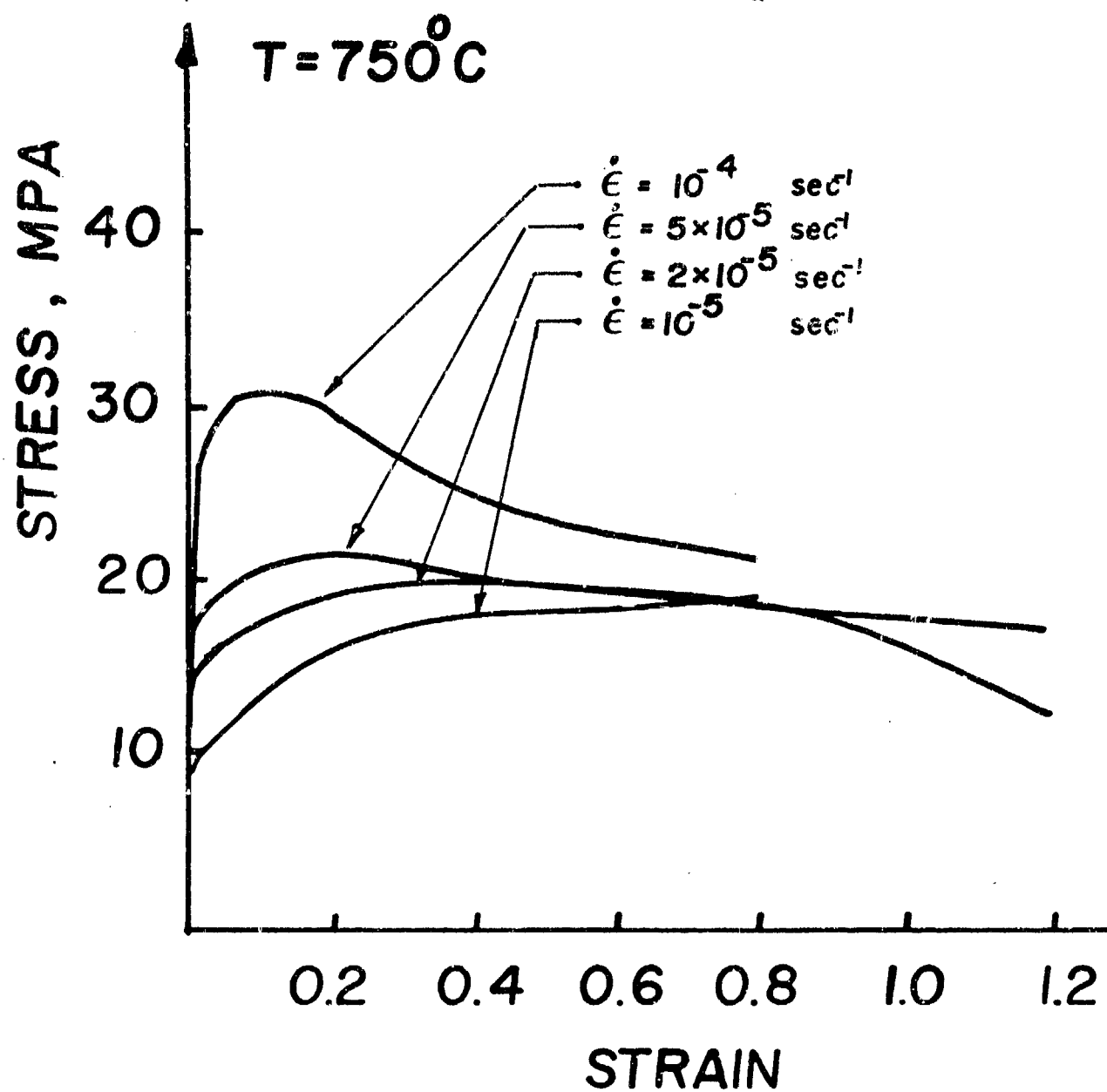


FIG. 1

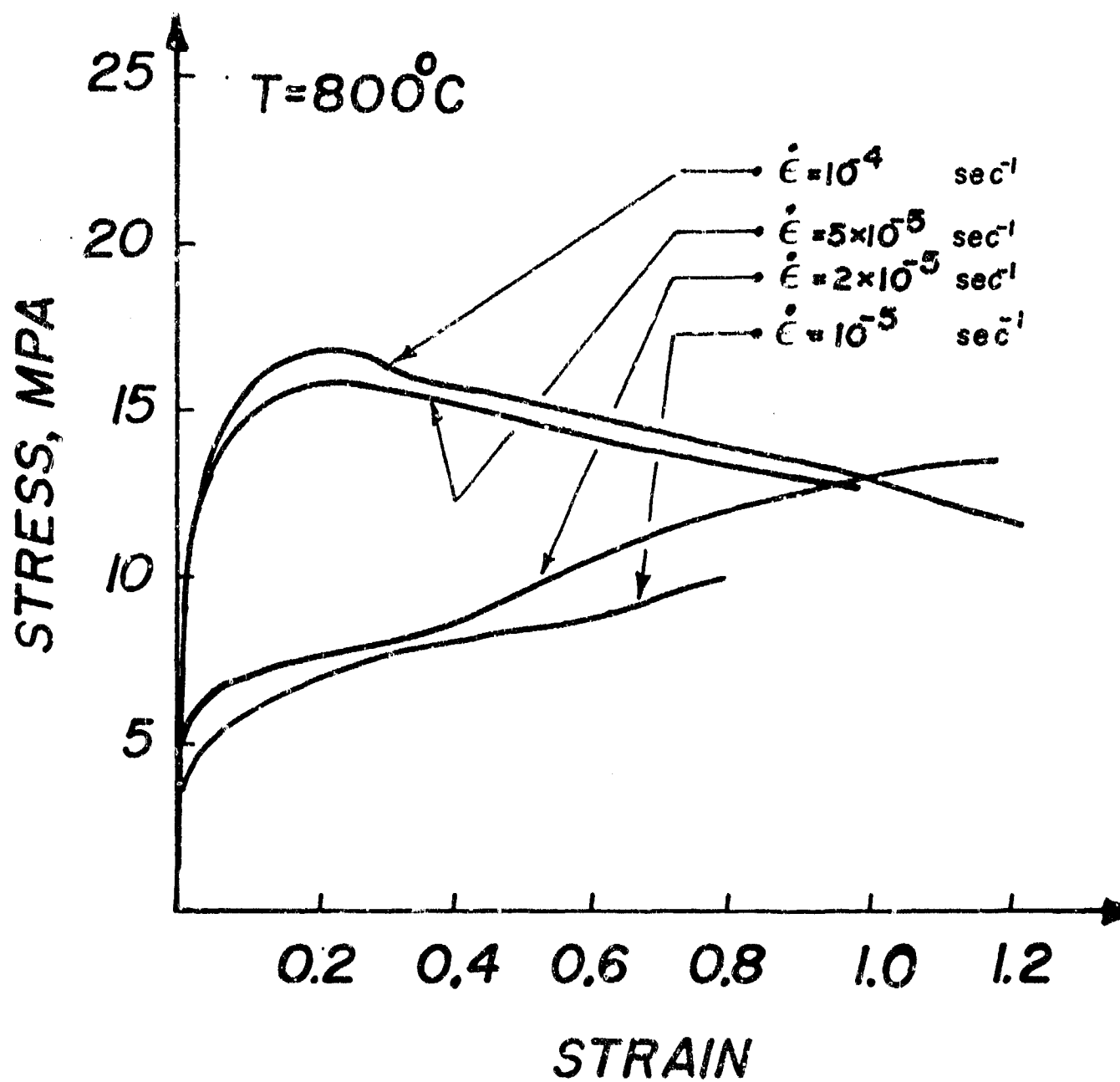


FIG. 2

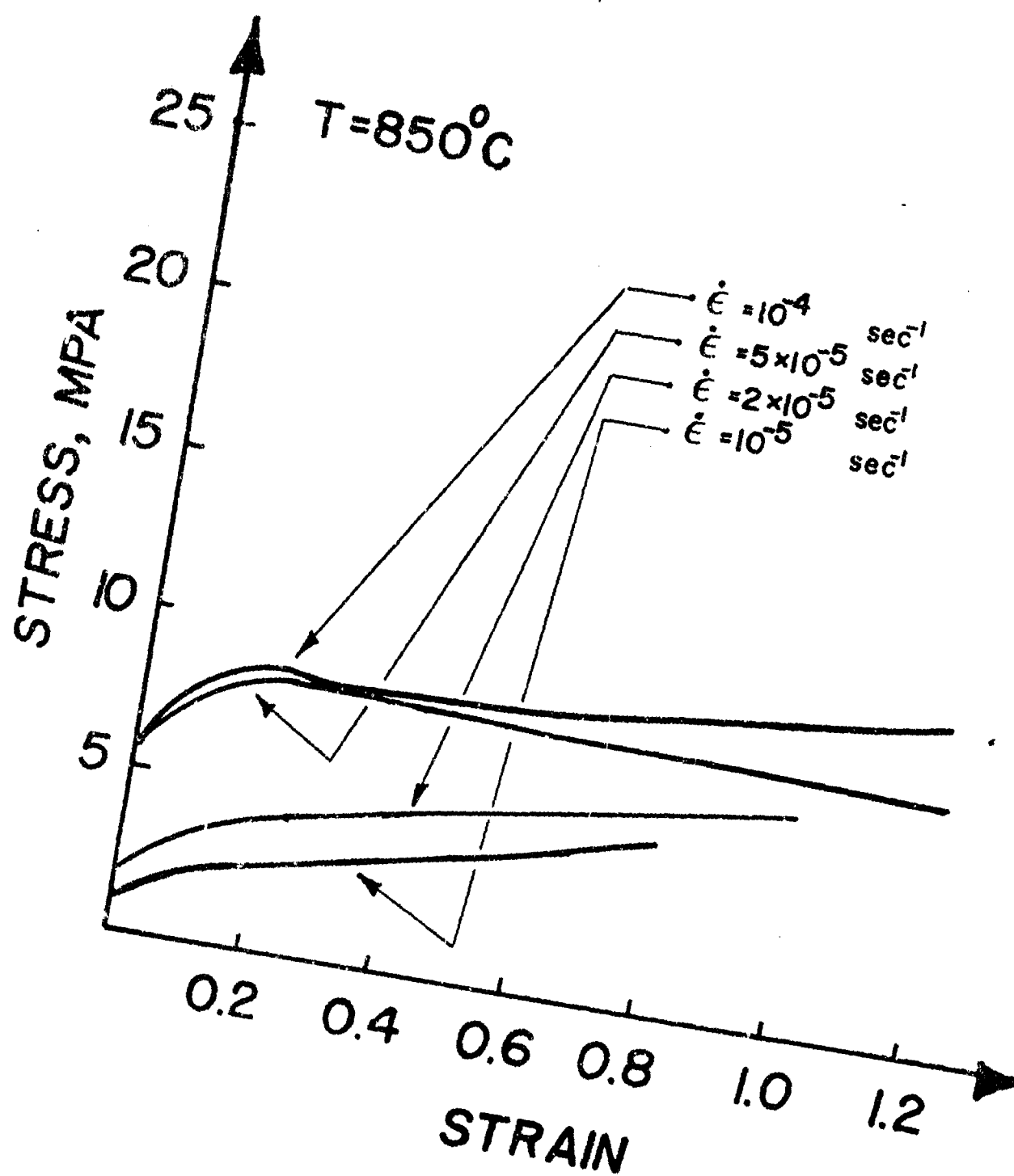


FIG. 3

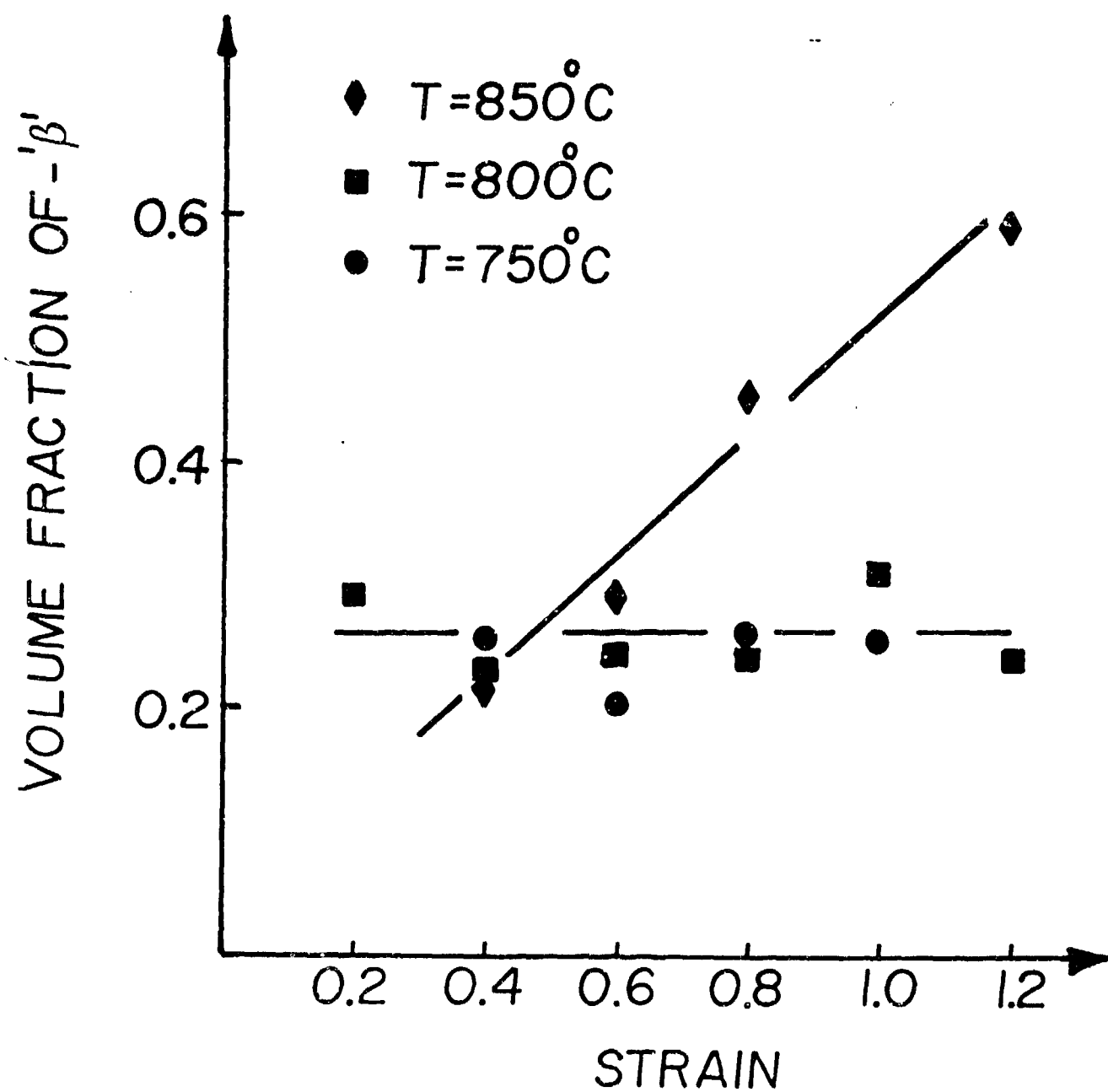


FIG. 4

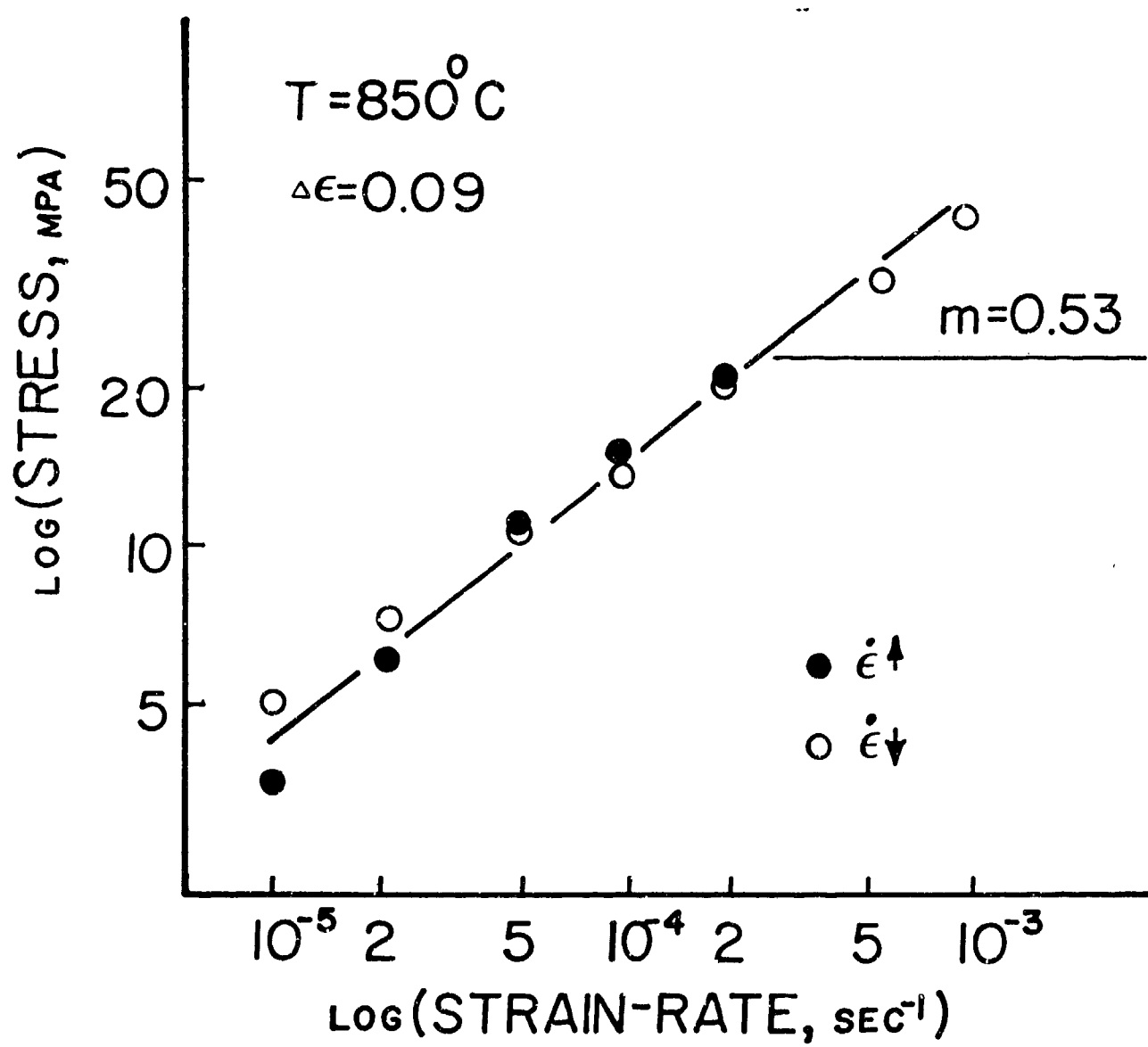


FIG. 5



Figure 6



Figure 7

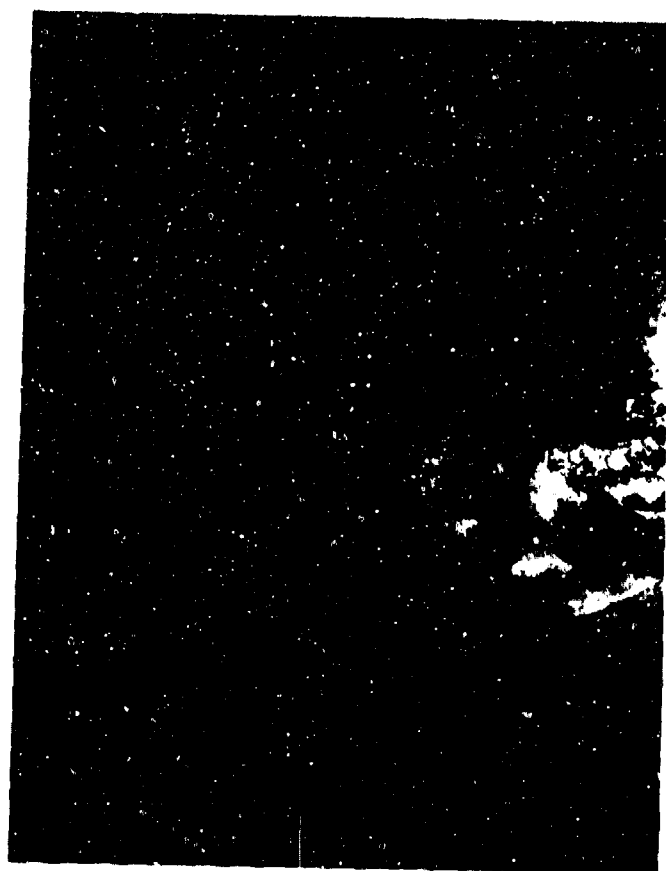


Figure 8

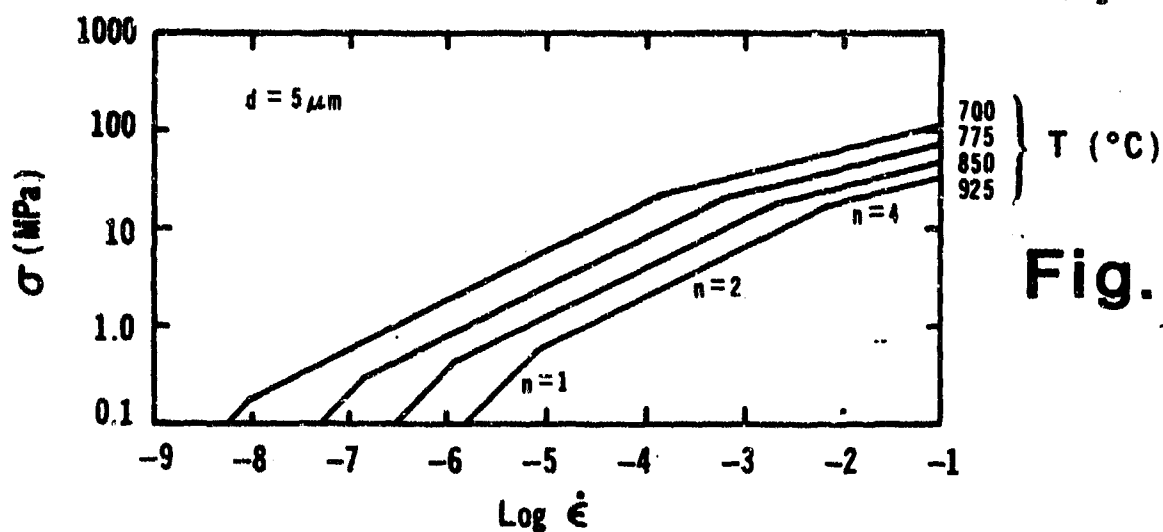


Fig. 9a

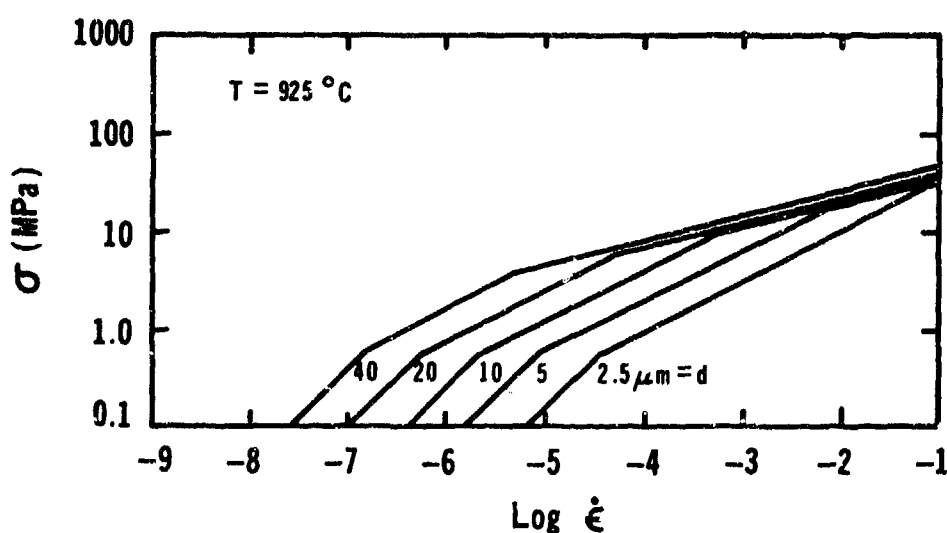
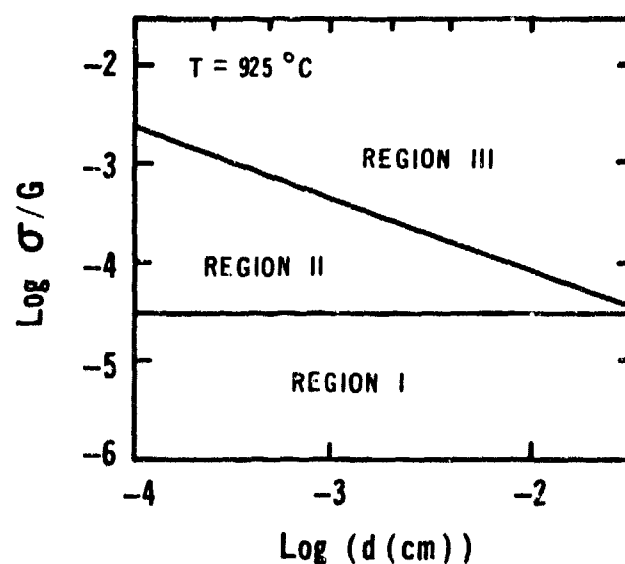
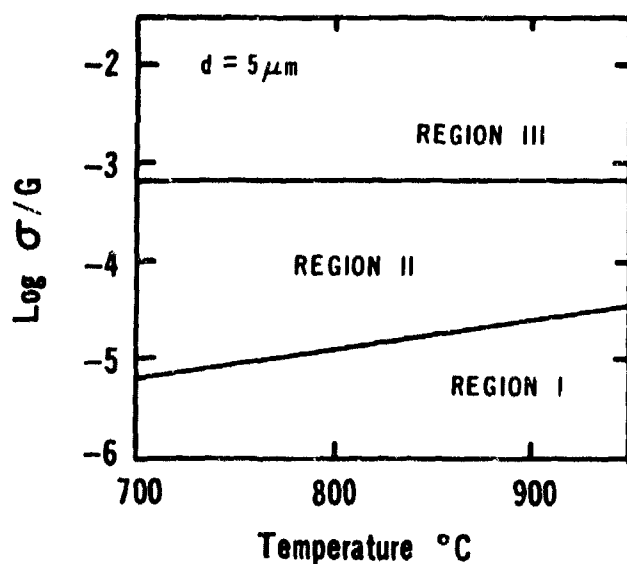
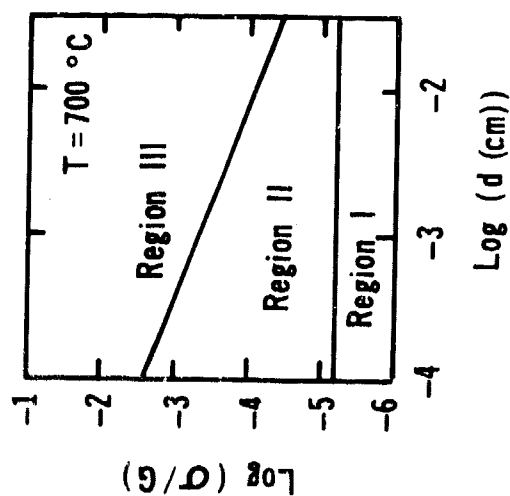
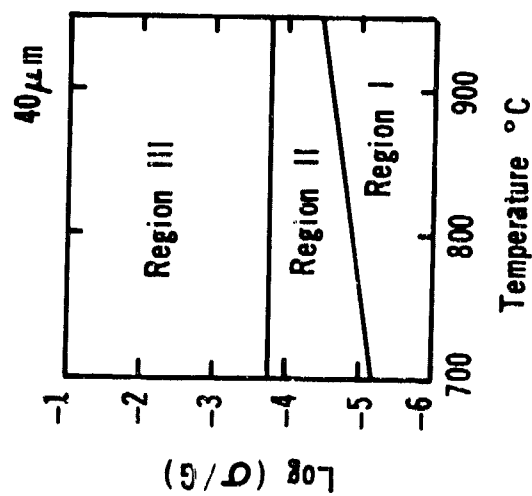
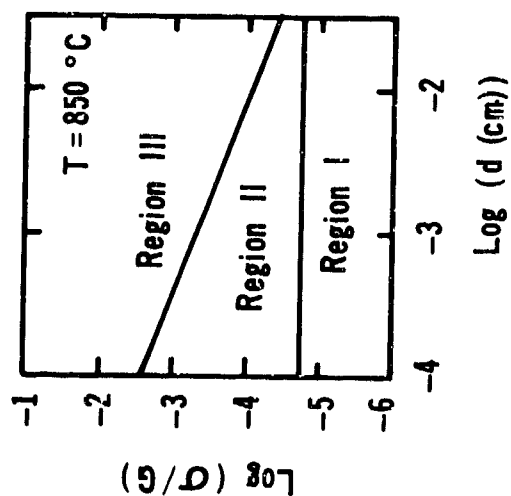
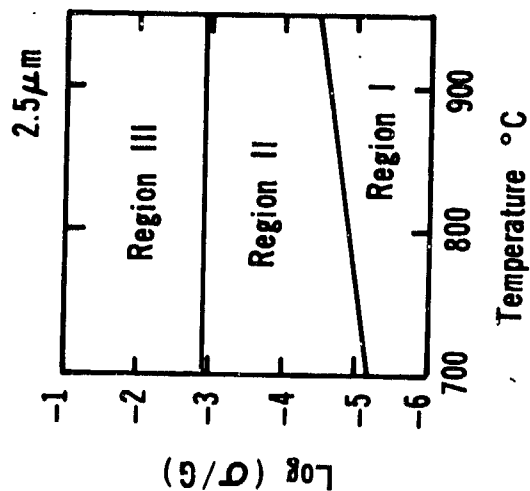
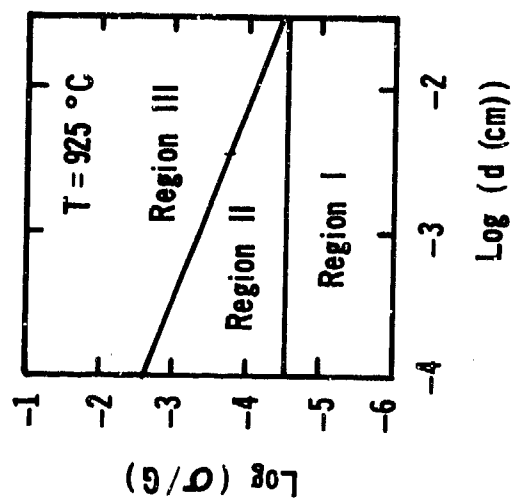
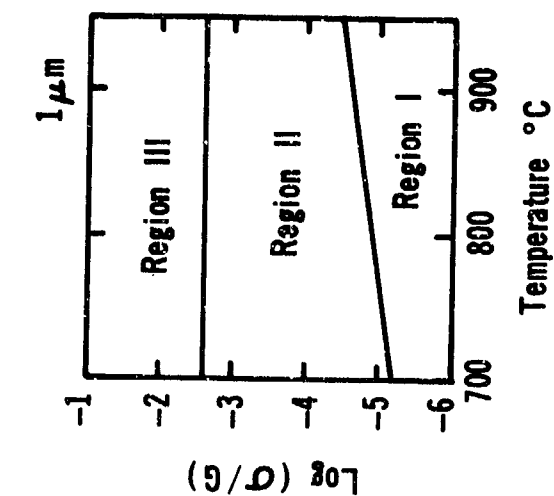


Fig. 9b





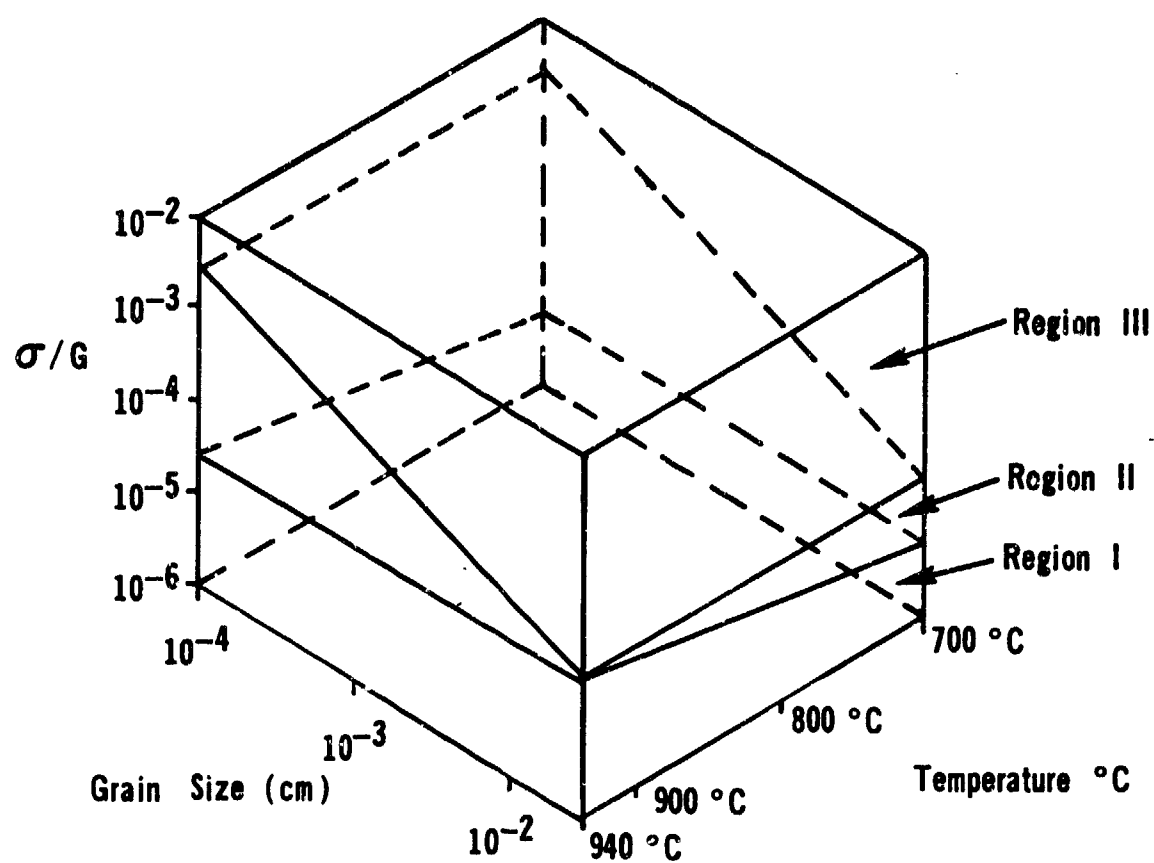


Fig. 11

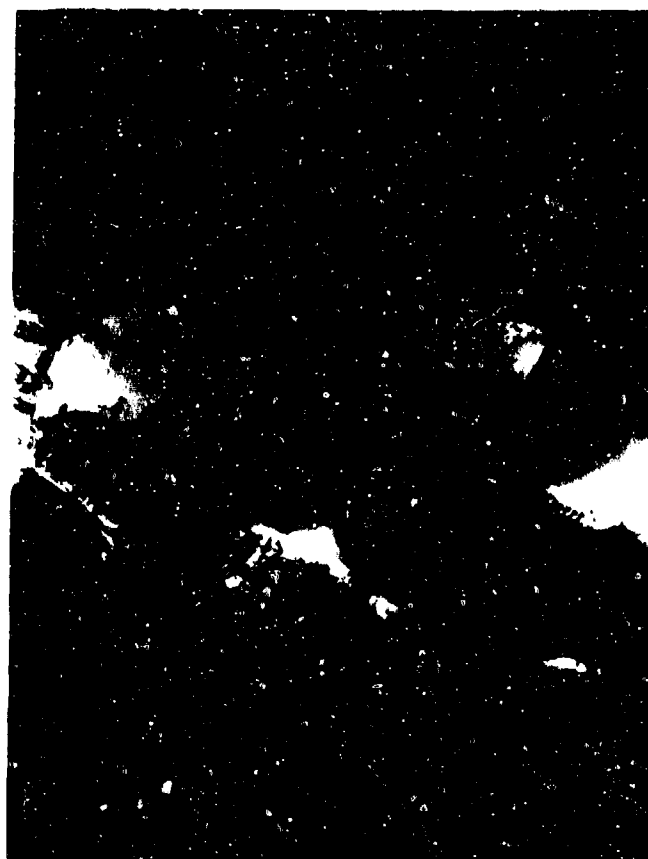


Figure 12

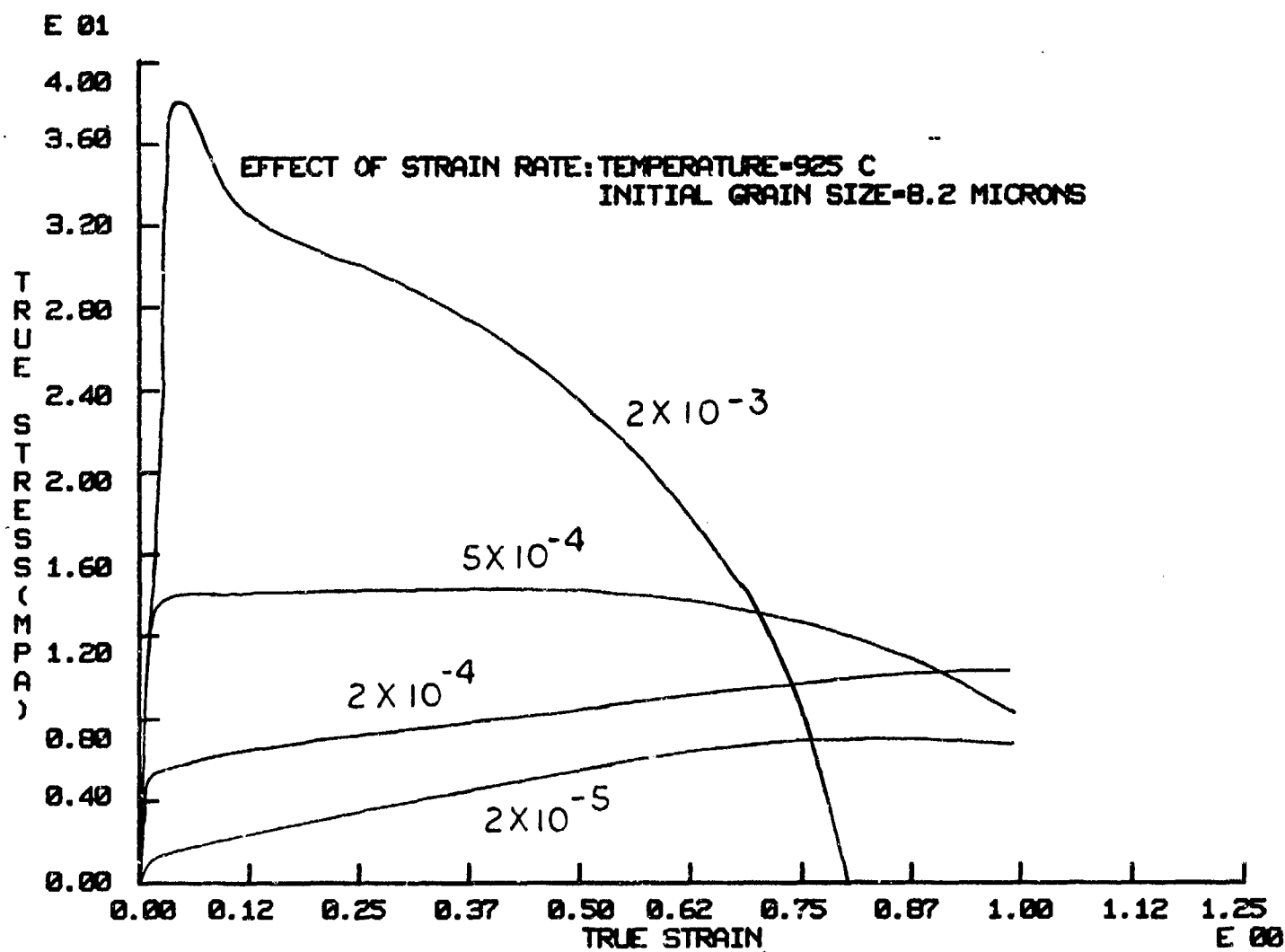


Figure 13

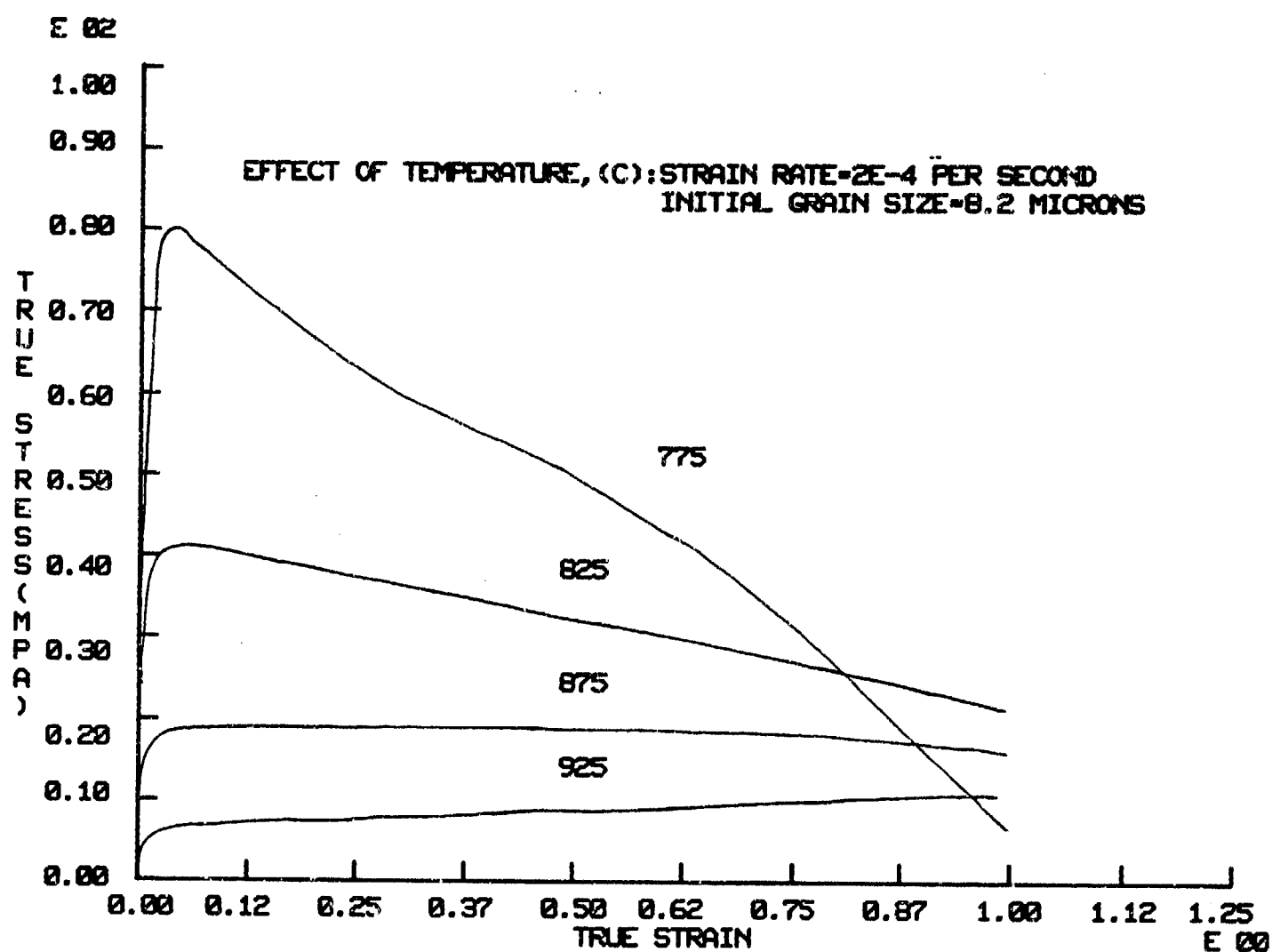


Figure 14

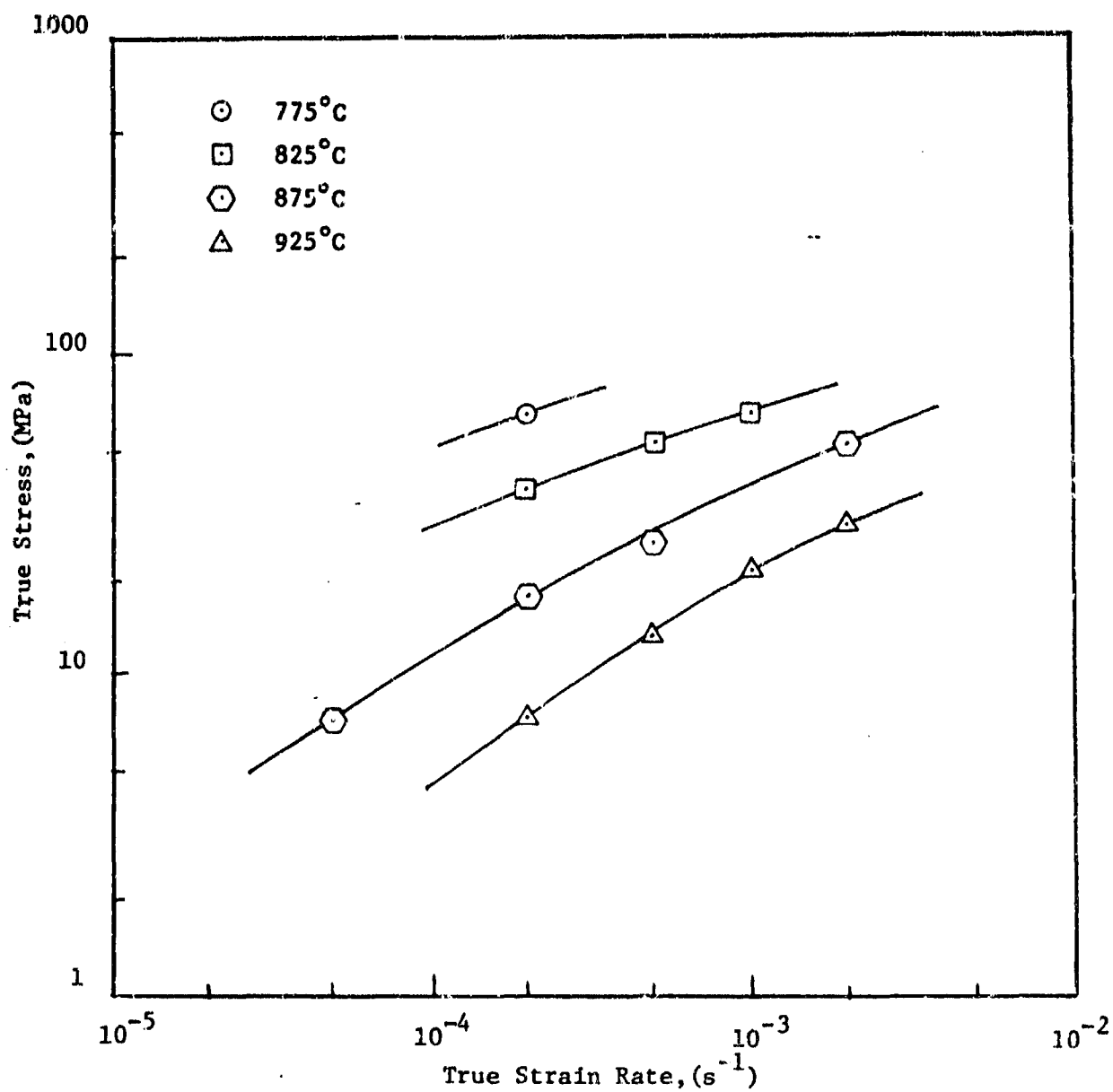


Figure 15 Log true stress-vs-Log true strain rate for the yield stress of Ti-6Al-4V with an initial grain size of 8.2 microns.

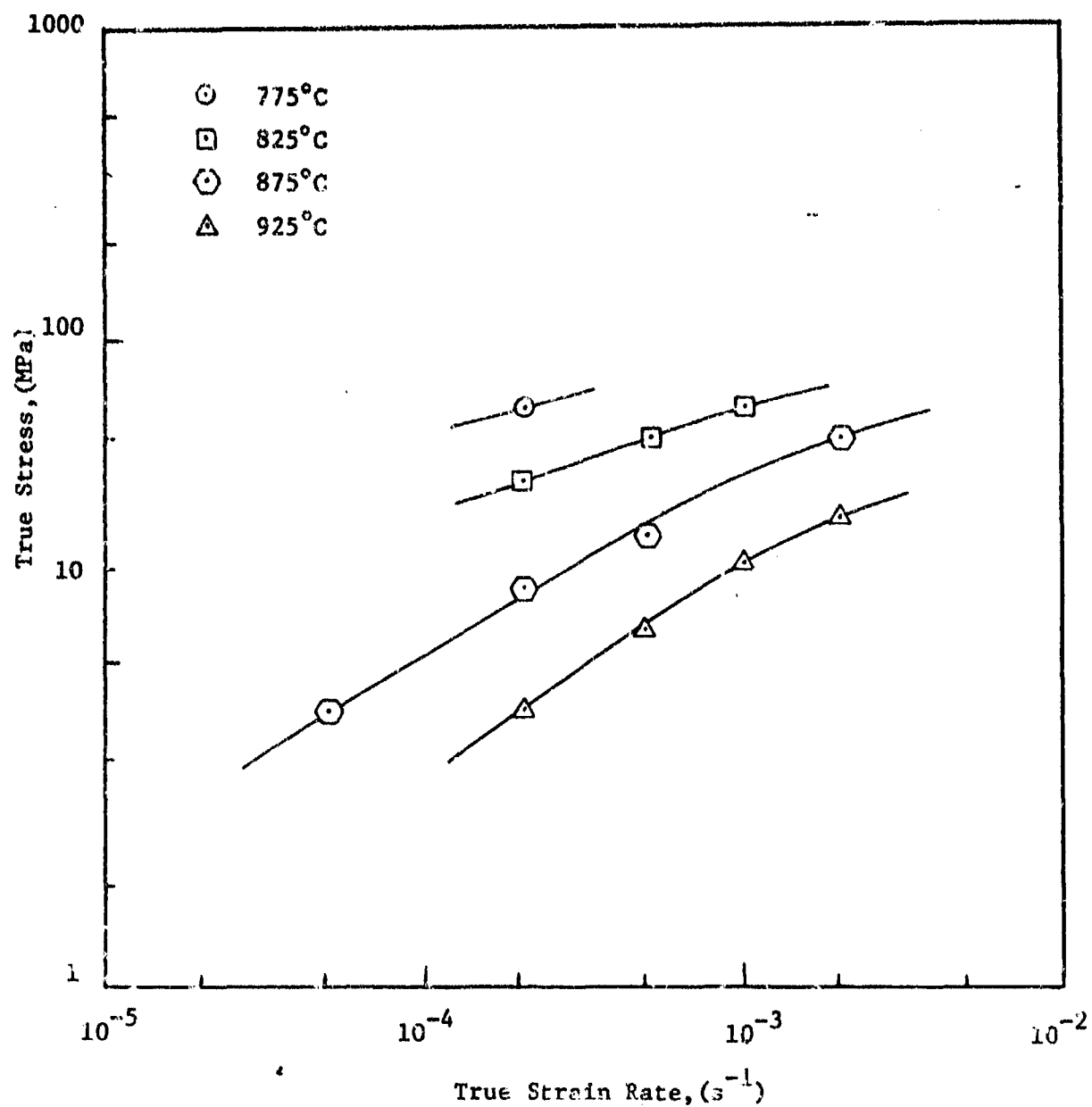


Figure 16 Log true stress-vs-Log true strain rate for Ti-6Al-4V at a true strain of 0.2. (Initial grain size is 8.2 microns)

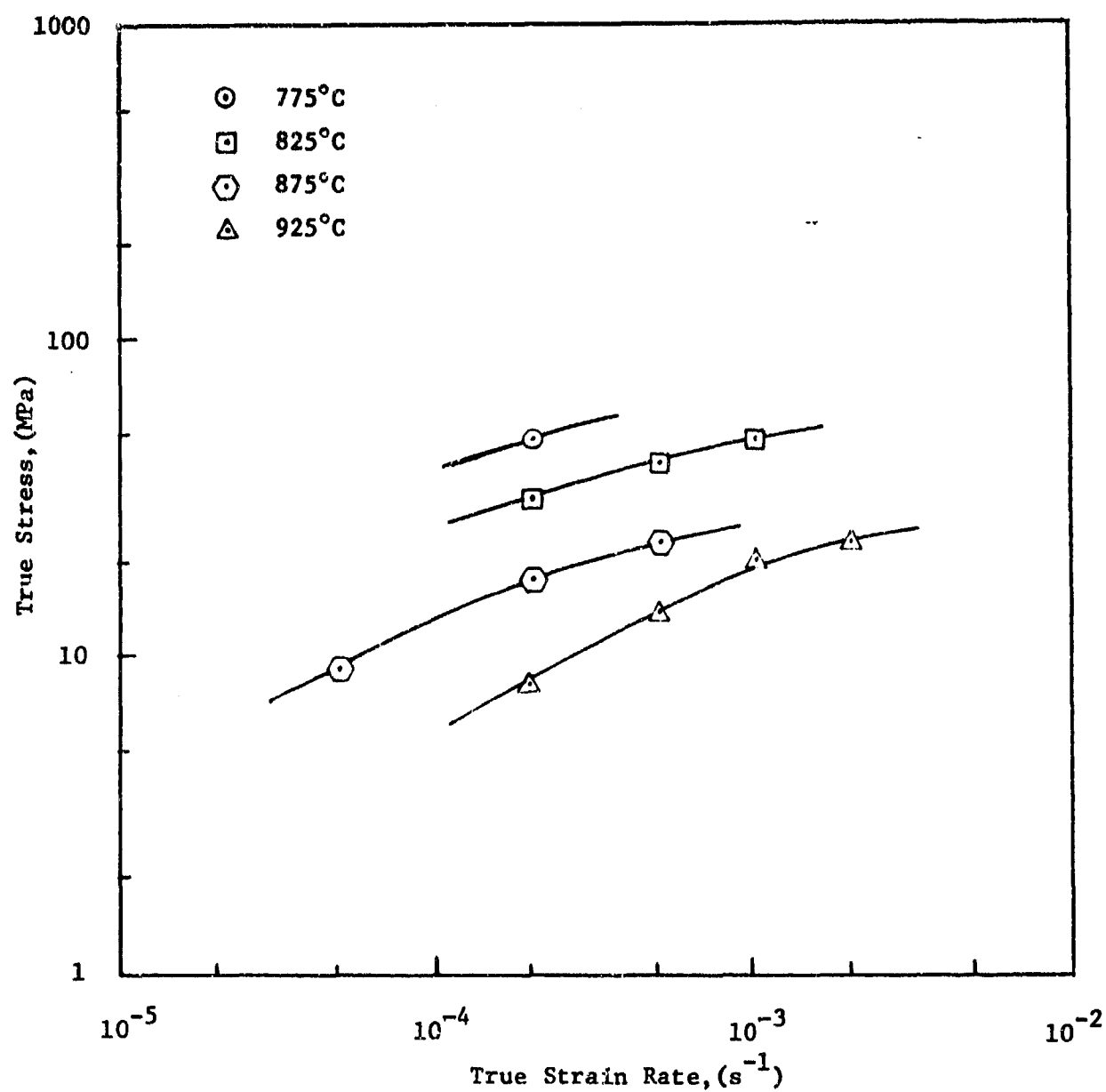
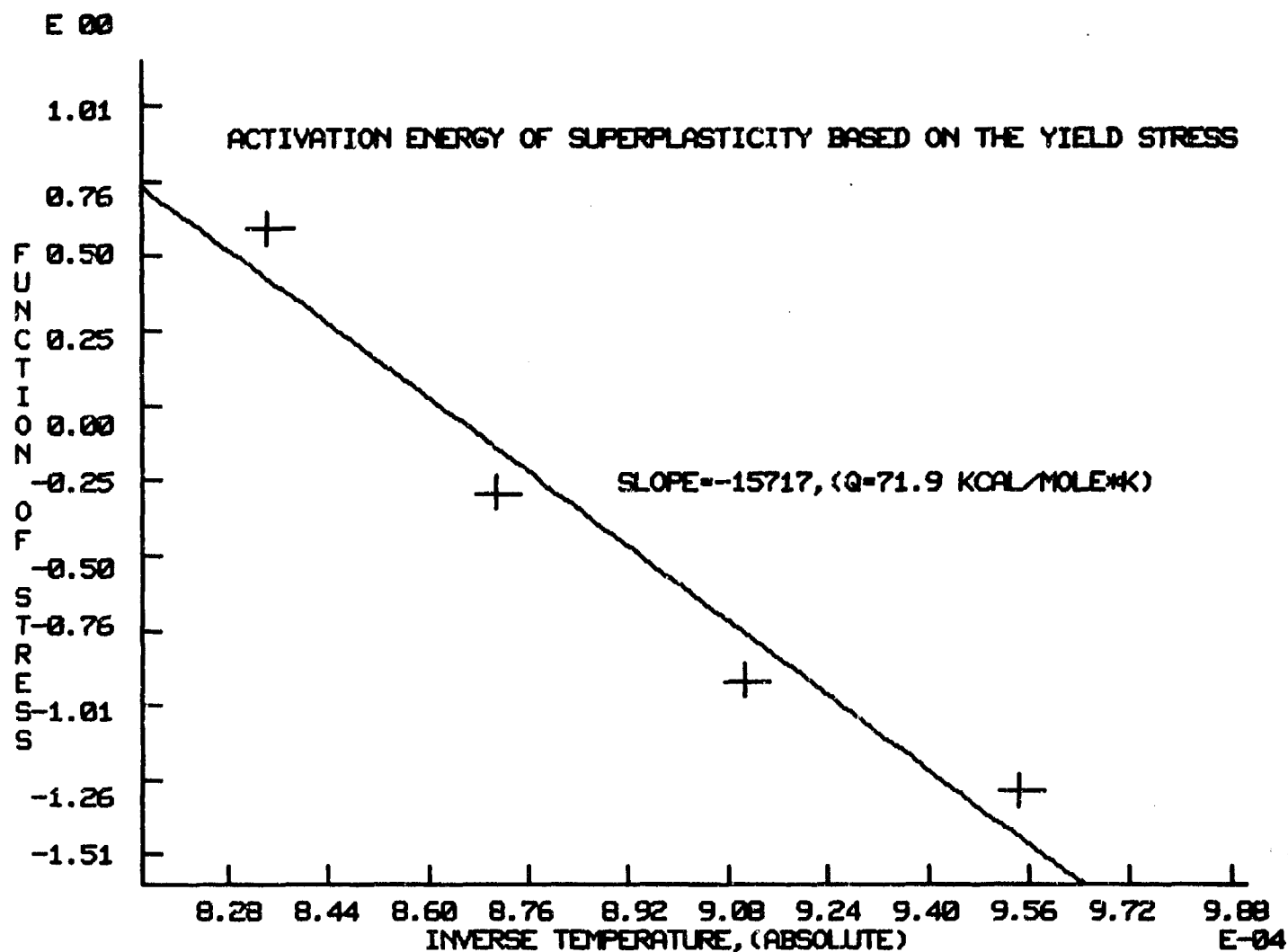


Figure 17 Log true stress-vs-Log true strain rate for Ti-6Al-4V with an initial grain size of 8.2 microns and at a true strain of 0.5.

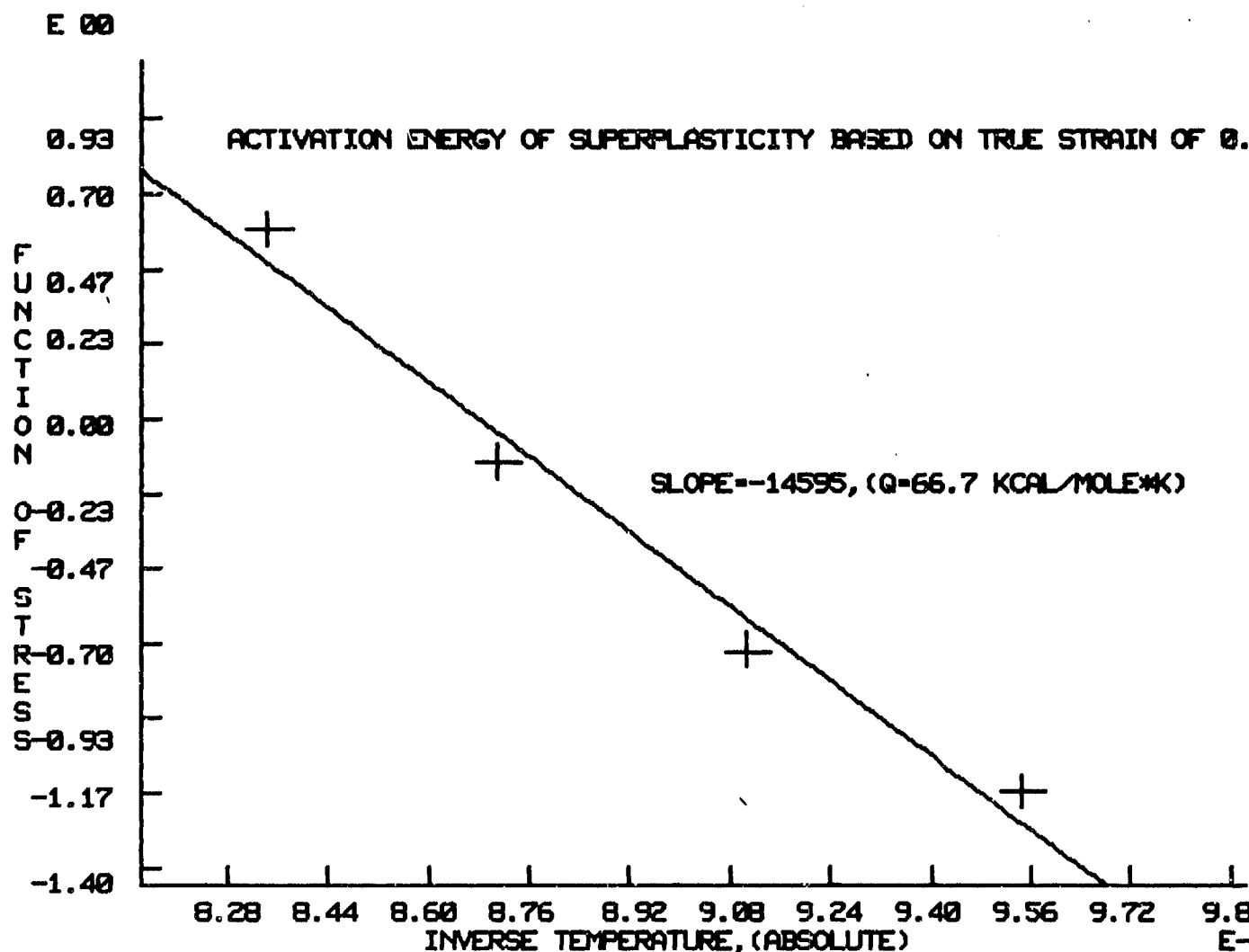


STRAIN RATE= 2.00000E-04 STRAIN= 0
 THE EQUATION FOR THE LINE IS: $Y = 13.545 + -15717.8 * (1/T)$
 THE COEFFICIENT OF DETERMINATION IS: .951409
 THE ACTIVATION ENERGY FOR CREEP IS: 71925.7 (CAL/MOLE*K)
 THE VALUE OF THE CONSTANT AT THE INTERCEPT IS 3.50724E+13
 THE STRAIN RATE SENSITIVITY IS 1.73

I	STRESS, (MPA)	T, (K)	X(I)	Y(I)
1	79.9	775	9.54198E-04	-1.30267
2	51.2	825	9.10747E-04	-.932951
3	23.1	875	8.71080E-04	-.299759
4	7.4	925	8.34725E-04	.590734

READY

Figure 18

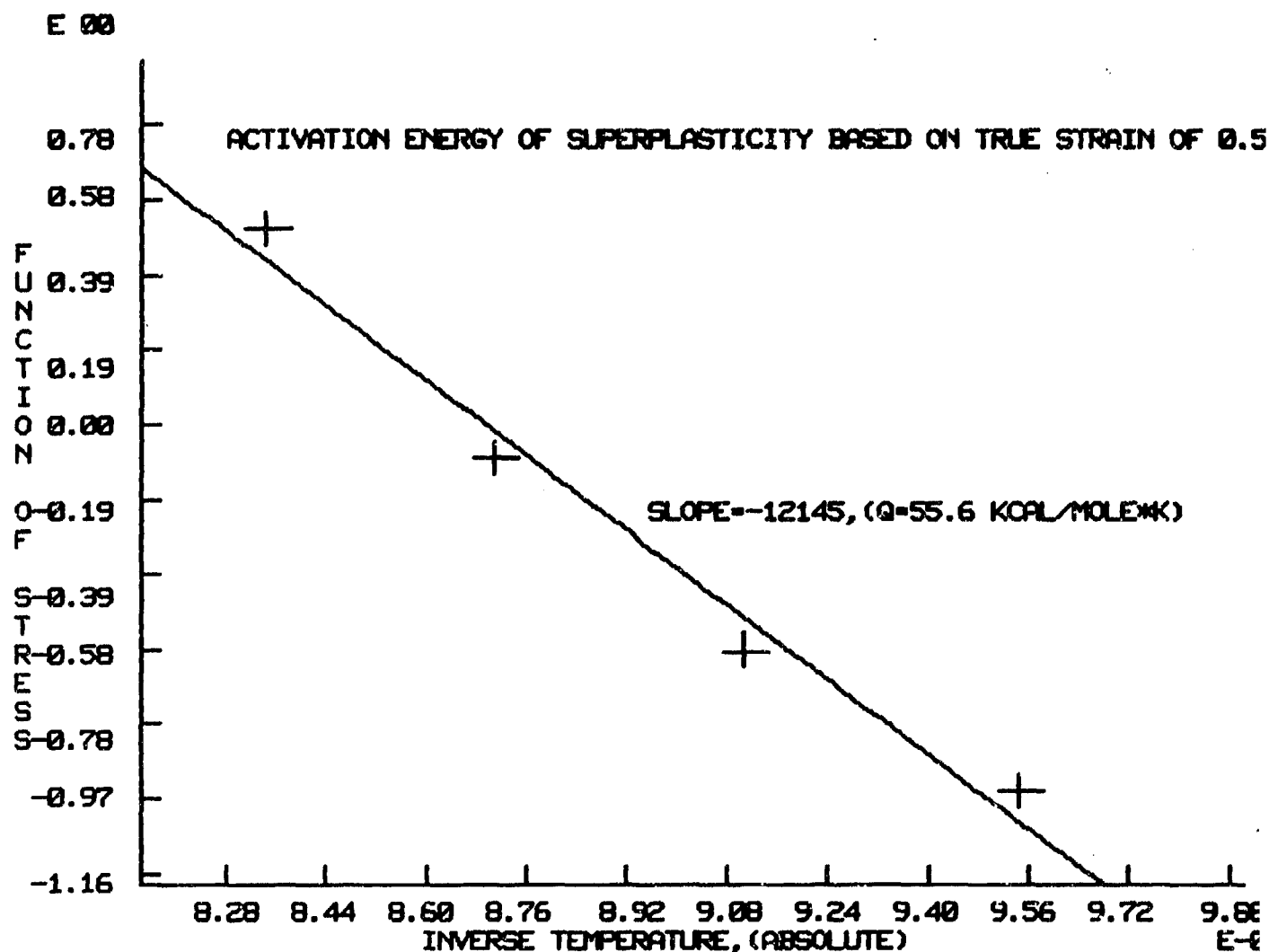


STRAIN RATE= 2.00000E-04 STRAIN= .2
 THE EQUATION FOR THE LINE IS: $Y = 12.6697 + (-14595.7) * (1/T)$
 THE COEFFICIENT OF DETERMINATION IS: .978184
 THE ACTIVATION ENERGY FOR CREEP IS: 66790.8 (CAL/MOLE*K)
 THE VALUE OF THE CONSTANT AT THE INTERCEPT IS 4.67451E+12
 THE STRAIN RATE SENSITIVITY IS 1.73

I	STRESS, (MPA)	T, (K)	X(I)	Y(I)
1	66.5	775	9.54198E-04	-1.16475
2	38.8	825	9.10747E-04	-.724593
3	18.6	875	8.71080E-04	-.136968
4	7.43	925	8.34725E-04	.587694

READY

Figure 19

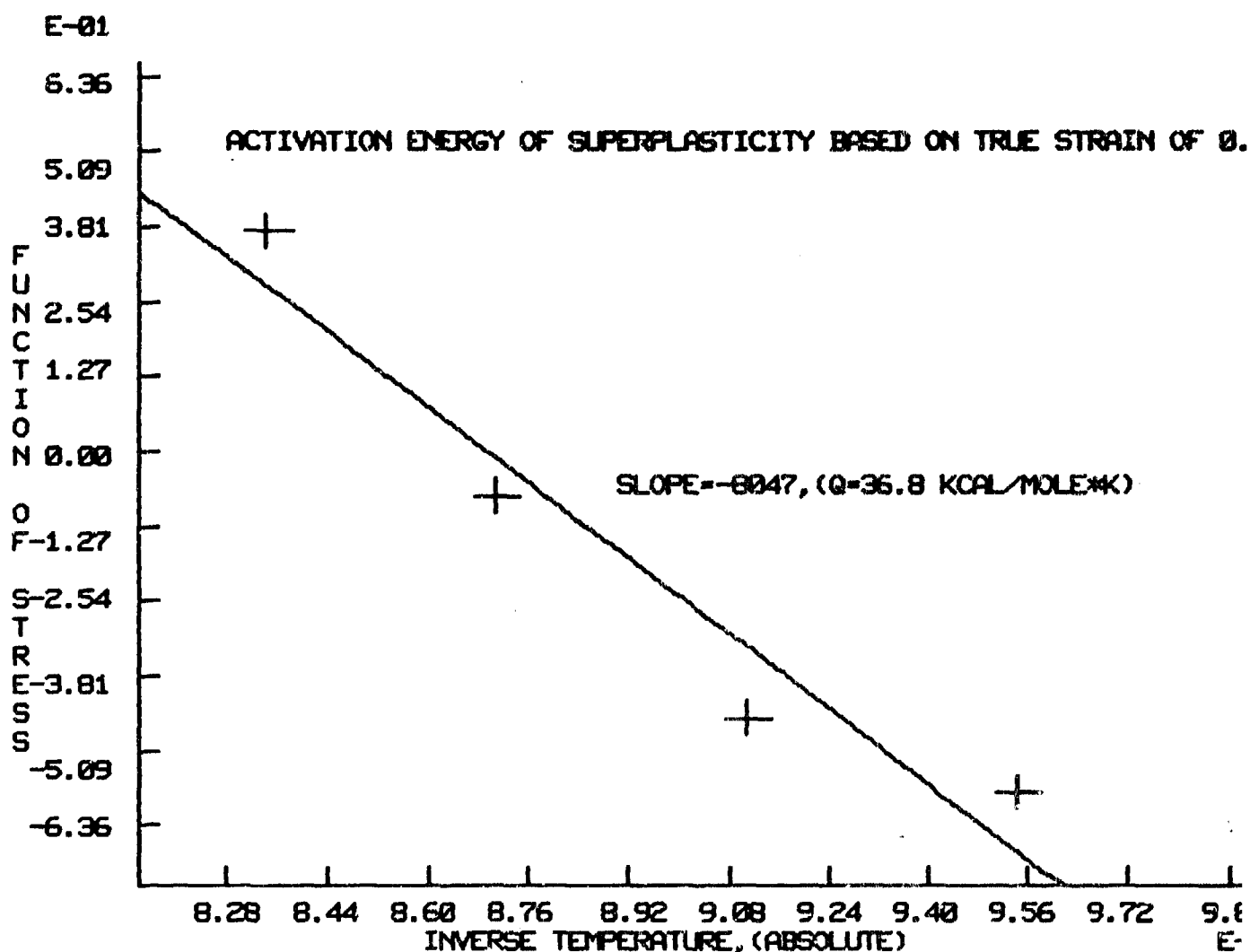


STRAIN RATE= 2.00000E-04 STRAIN= .5
 THE EQUATION FOR THE LINE IS: $Y = 10.5624 + -12145.1 * (1/T)$
 THE COEFFICIENT OF DETERMINATION IS: .979595
 THE ACTIVATION ENERGY FOR CREEP IS: 55576.6 (CAL/MOLE*K)
 THE VALUE OF THE CONSTANT AT THE INTERCEPT IS 3.65095E+10
 THE STRAIN RATE SENSITIVITY IS 1.73

I	STRESS, (MPA)	T, (K)	X(I)	Y(I)
1	50	775	9.54198E-04	-.950482
2	32.2	825	9.10747E-04	-.584505
3	17.4	875	8.71080E-04	-.086861
4	8.3	925	8.34725E-04	.5045

READY

Figure 20



STRAIN RATE= 2.00000E-04 STRAIN= .75
 THE EQUATION FOR THE LINE IS: $Y = 6.99801 + -8047.24 * (1/T)$
 THE COEFFICIENT OF DETERMINATION IS: .931292
 THE ACTIVATION ENERGY FOR CREEP IS: 36824.7 (CAL/MOLE* K)
 THE VALUE OF THE CONSTANT AT THE INTERCEPT IS 9.95424E+06
 THE STRAIN RATE SENSITIVITY IS 1.73

I	STRESS, (MPA)	T, (K)	X(I)	Y(I)
1	30.6	775	9.54198E-04	-.581562
2	27.1	825	9.10747E-04	-.454951
3	17.2	875	8.71080E-04	-.0781749
4	9.9	925	8.34725E-04	.372056

READY

Figure 21

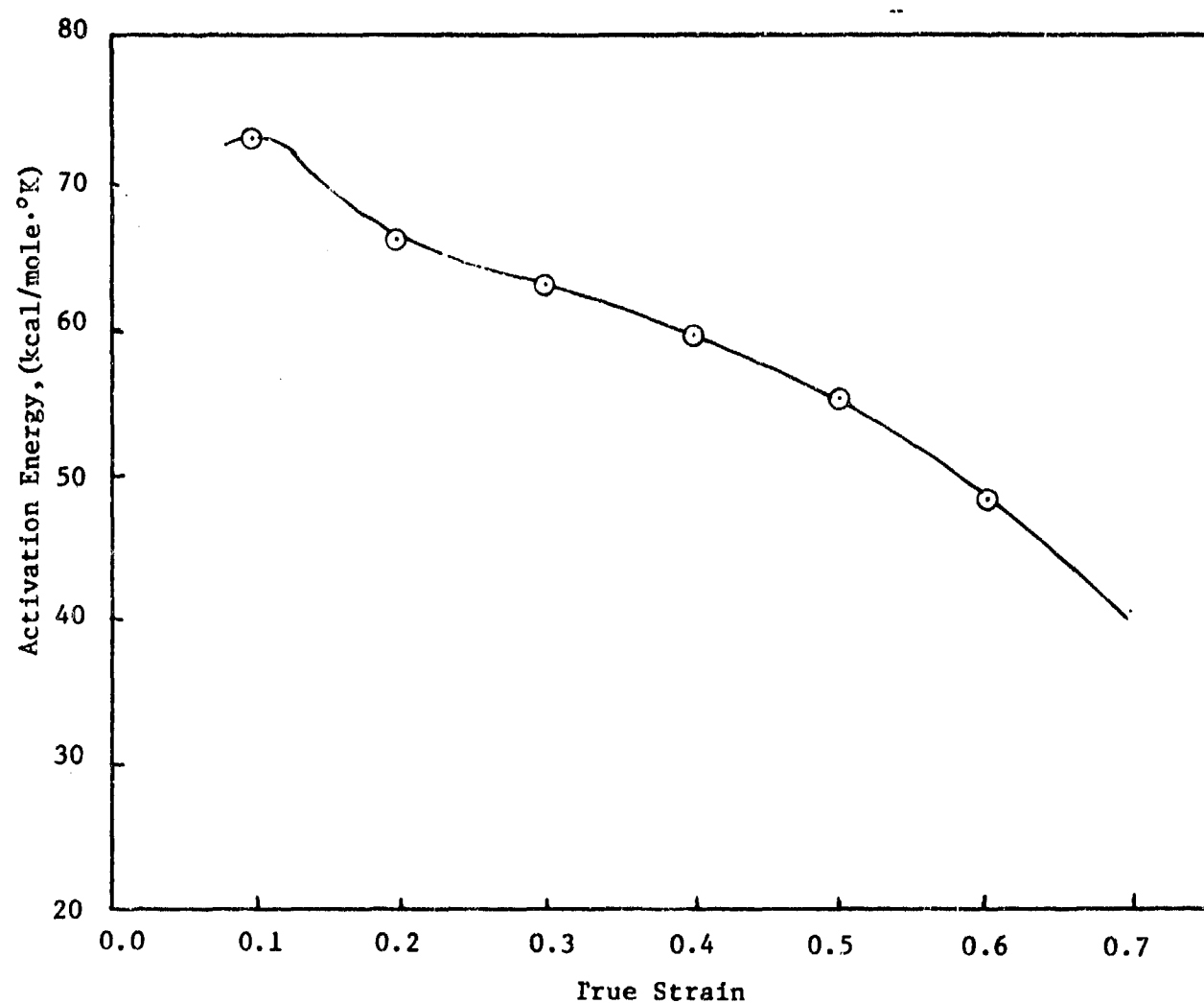


Figure 22 Variation of activation energy with strain in Ti-6Al-4V, initial grain size is 8.2 microns, true strain rate is 2×10^{-4} .

List of dissertations in Preparation

1. Mechanical Properties and Cavitation in Ti-6Al-4V Alloy During Superplastic Deformation (Ph.D.: Giora Gurewitz).
2. Micromechanisms for Deformation and Deformation and Deformation Mechanism Maps in Ti-6Al-4V Alloy (M.S.: Mike Meier).

List of Personnel Involved in Research

1. Giora Gurewitz (Ph.D. candidate)
2. Mike Meier (M.S. candidate)
3. Professor Amiya Mukherjee (Principal Investigator)

List of Compling Activities

1. Dr. C. H. Hamilton and Dr. A. K. Ghosh, Science Center. North American Rockwell, Thousand Oaks, California.
2. Dr. A. Arieli and Dr. R. Vastava, Northrop Corporation, Airplane Division, Hawthorne, California.